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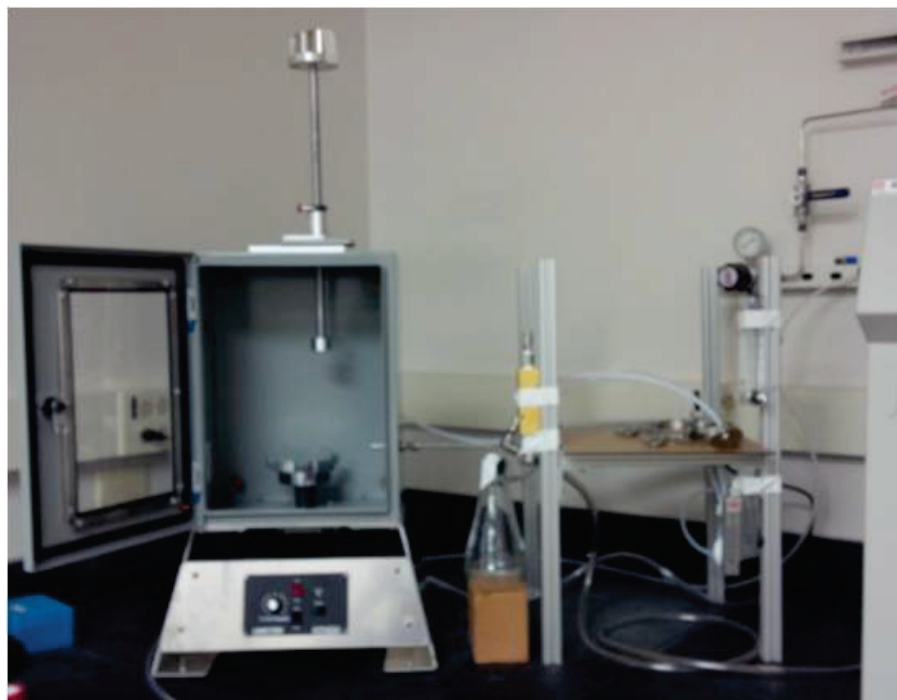
Environmental Consequences of Nanotechnologies

Abrasion Testing of Products Containing Nanomaterials, SOP-R-2

Scientific Operating Procedure Series: Release (R)

Monica A. Ramsey, Jonathon A. Brame, Aimee R. Poda,
Michael Cuddy, and Robert D. Moser

April 2016



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Abrasion Testing of Products Containing Nanomaterials, SOP-R-2

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Final report

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Abstract

Abrasion is an important consideration for the potential release of nanoparticles during the service life of a nanotechnology. This SOP presents a general method for abrading a material using a rotating sample platform in contact with a weighted sandpaper fixture. Particles collected with this method are analyzed with a condensation particle counter (CPC) and a fast mobility particle sizer (FMPS), with additional particles collected on in-line filters for further analysis. The abrading process is carried out in a particle-free environment and released particles are moved by a defined airflow from the chamber to the collection/analysis apparatus. Particle release is monitored in real time by the CPC (10nm - >1 μ m) and FMPS (32 particle size bins from 5-550 nm), and further characterization of filter-collected particles can be accomplished post-testing.

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Contents

Abstract.....	ii
Figures	iv
Preface	v
Acronyms	vi
1 Introduction.....	1
2 Background.....	2
3 Scope	5
4 Terminology.....	6
Related Documents.....	6
Definitions.....	6
5 Materials and Apparatus	8
Materials.....	8
Apparatus	8
6 Procedure	10
Specimen Preparation	10
Analysis	11
7 Reporting.....	12
Analysis of Results	12
Key Results Provided.....	12
QA/QC Considerations	14
References.....	15
Appendix: Notes and Supplementary Data.....	17
Report Documentation Page	

Figures

Figures

Figure 1. Flowchart showing testing procedure to prepare and abrade a sample specimen to show abrasion-induced release.....	5
Figure 2. Experimental setup for sanding simulation system and sampling monitoring system.	9
Figure 3. Total particle concentration released during abrasion of an ABS 3-D printed puck with Bronze nano-colorants, as measured by CPC. Abrasion started at t=60s and concluded at t=300s. Solid line shows average particle counts with thin lines representing \pm standard deviation from three replicate tests.	13
Figure 4. Particle size distribution for abrasion of an ABS 3-D printed puck with nano-colorants, measured by FMPS. Data shows that the majority of particles fall in the 5-50 nm size range.....	13

Preface

This procedure was developed under the Engineer Research Development Center (ERDC) Environmental Quality and Technology (EQT) Research Program titled “Environmental Consequences of Nanotechnologies.” Procedures link to the ERDC NanoGRID (Guidance for Risk Informed Deployment) framework for testing the exposure and hazard of nanotechnology Environmental Health and Safety (EHS). The technical monitor was Dr. Elizabeth Ferguson.

The work was coordinated by the Environmental Chemistry Branch (CEERD-EPC) of the Environmental Processes and Engineering Division (CEERD-EP) at the U.S. Army Engineer Research and Development Center – Environmental Laboratory (ERDC-EL). David Morrow was the Branch Chief, CEERD-EPC; Warren P. Lorentz was the Division Chief, CEERD-EP; and Dr. Elizabeth Ferguson was the Technical Director for Military Environmental Engineering and Sciences. The Deputy Director of ERDC-EL was Dr. Jack Davis and the Director was Dr. Beth Fleming.

COL Bryan S. Green was the Commander of ERDC, and Dr. Jeffery P. Holland was the Director.

Acronyms

ANOVA	Analysis of Variance
CPC	Condensation Particle Counter
CNTs	Carbon Nanotubes
DRI	Direct Reading Instruments
EDS	Energy Dispersive X-ray Spectroscopy
EM	Electron Microscopy
FFF	Field Flow Fractionation
FMPS	Fast Mobility Particle Sizer (also SMPS-Scanning Mobility Particle Sizer)
HEPA	High-Efficiency Particulate Air
HHPC	Hand-Held Particle Counter
LM	Light Microscopy
Lpm	liters per minute
M	Mass
MC	Mass Concentration
NC	Number Concentration
NP	Nanoparticle
NRP	Number of Released Particles
OPC	Optical Particle Counter
PM	Particulate Matter
PSD	Particle Size Distribution
PVC	Poly Vinyl Chloride
SA	Surface Area
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscope
WG	Water Gauge

1 Introduction

This SOP describes how to detect and quantify the release of nanoparticles from surface coatings into the air using a mechanical process that employs abrasion to simulate sanding. A material containing nanoparticles will be physically abraded and the materials released will be collected in a custom abrasion testing system. They will then be characterized by different methods such as Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) and other methods.

2 Background

Several recent studies have attempted to bridge the critical knowledge gap of nanomaterial release from nano-composite materials using mechanical abrasion techniques. Hirth et al. showed that release of carbon nanotubes (CNTs) from a nanocomposite material required a combination of UV-induced weathering and physical abrasion (Hirth et al. 2013). Several other studies found some level of individual nanomaterial release under heavy mechanical stress (Golanski et al. 2012, Huang et al. 2012, Schlagenhauf et al. 2012, Fiorentino et al. 2015), while others found no individual nanomaterials released under similar mechanical stress conditions (Bello et al. 2009, Vorbau et al. 2009, Göhler et al. 2010, Cena and Peters 2011). Many of these studies identified difficulty in generating reproducible results during these mechanical abrasion studies (Vorbau et al. 2009, Göhler et al. 2010, Golanski et al. 2012) due to variability in material, abrasion and aerosol sampling as a significant issue in determining release of engineered nanomaterials (ENMs). Even when nano-sized particles are identified in a particle-size distribution, it can be difficult to determine whether they are ENMs themselves, ENMs included in a composite formulation, or simply small particles of the composite matrix released during abrasion (Dylla and Hassan 2012).

These difficulties point to the necessity of identifying a standardized procedure for abrasion testing of ENM-containing materials. One of the most common techniques relies on adaptations to the Taber abraser, a sanding simulation device. The test is described in many national and international standards, including ASTM D 4060-95: 2007 and ASTM C1353-07, ISO 5470-1:1999, and DIN 53754:1977. The standard test is performed with the sample being rotated while in contact with two abrasive wheels moving in the opposite direction. Particle release depends on the surface coating and substrate material used.

Adaptations to this test method can be used with a range of nanomaterial/polymer products in which sand paper and samples are independently rotated or held in a stationary position. There are optional adjustments that can be made to add variation by changing wheel types, adding or removing a normal force in the range of 2.5-10N, or adjusting the number of abrasive cycles. Also, the type, kind, or amount of finishing materials, grit of sandpaper, size of sandpaper, and sample disks, time of contact, and mounting or tension of the specimen can affect the resistance of the

abrasion. All of these variables influence the number concentration, respirable mass concentration, and size distribution of airborne particles released.

For weak shear forces, an increase in nanoparticle number concentration above background was found when composites were subjected to simulated wear with the Taber abraser (Huang et al. 2012). However, no significant particle release compared to background was observed in another Taber abraser study (Wohlleben et al. 2013). Sanding with a miniature sander showed a substantial increase in airborne particle number concentrations in one study (Golanski et al. 2011), while in a field study, manual sanding of CNT-epoxy nanocomposite increased mass but not number concentration of particles compared to the background (Göhler et al. 2010).

A study performed on a do-it-yourself sanding process with matrix materials containing nanofillers led to the conclusion that the rigidity of matrix, rather than the presence of nanofillers, played a dominant role in determining the particle mass and size distribution of released aerosols (Wohlleben et al. 2011, Golanski et al. 2012). This outcome was similar to the conclusion that nanoparticle emissions from surface coatings via a miniature sander depended largely on the coating material rather than the presence of nanomaterial (Golanski et al. 2011).

After particles are released, proper characterization is essential to determine the potential hazard of ENMs that may be included in the released material. The particle number density is characterized with a condensation particle counter (CPC), while a fast mobility particle sizer (FMPS) determines the particle size/mass distribution. Light microscopy (LM), scanning electron microscopy (SEM), and transmission electron microscope (TEM) are three techniques to further analyze the characterization of the wear particles. These procedures can also be coupled with energy dispersive x-ray spectroscopy (EDS) for chemical microanalysis.

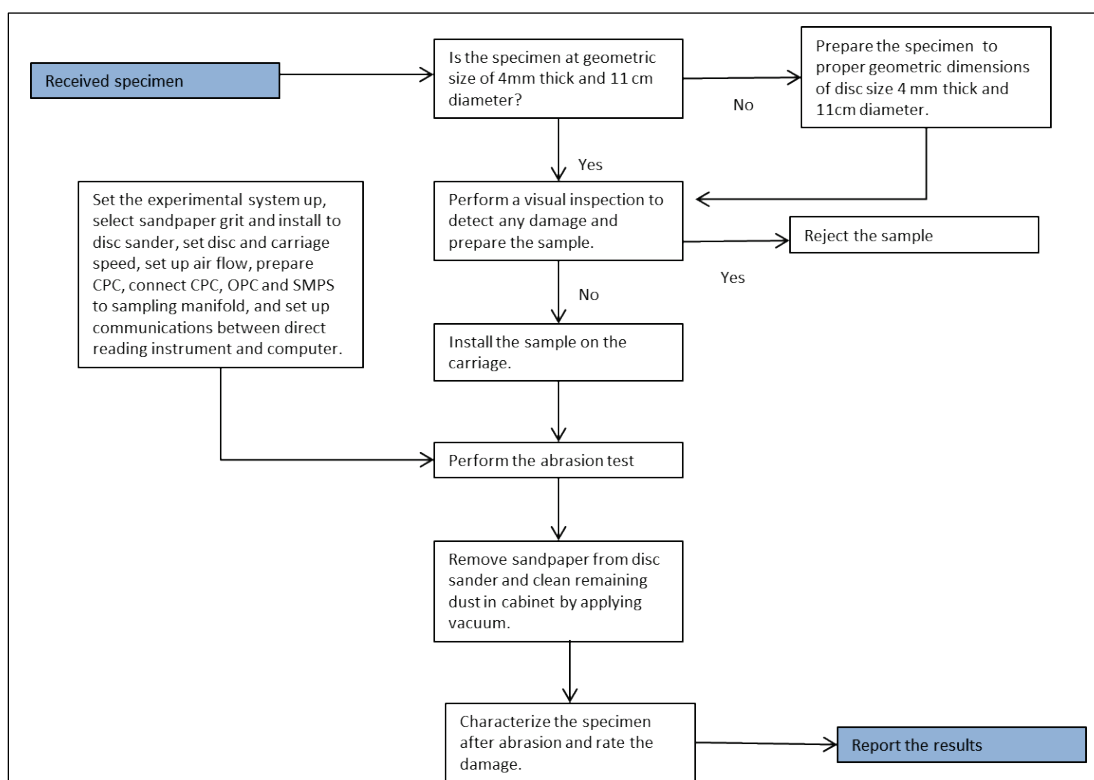
One advantage of abrasion testing is it can provide data in a matter of minutes compared to the years that may be required by in-use testing. The results can be used to inform risk decisions based on the mass of material released, the number of nanoparticles, the size and composition of the particles, and the rate of their release. There are some limitations associated with abrasion testing, such as limited shear rates, potential

clogging of the abrasion material due to pickup of test material, and potential contamination of testing due to interference by abrading material (e.g., an Al_2O_3 wheel). However, these limitations can be overcome significantly through test design and operational practices to ensure that the data represents realistic release scenarios.

3 Scope

This SOP is used to investigate the emission of airborne particles generated by abrasion testing of products containing nanomaterials. This test will physically abrade the surface of coatings or bulk materials that contain nanomaterials. Possible applications include CNTs incorporated in epoxy or other polymer matrices and TiO₂ embedded in concrete. The particle number/mass concentration, particle size distribution, and airborne particle morphology under different test conditions can be determined using this technique.

Figure 1. Flowchart showing testing procedure to prepare and abrade a sample specimen to show abrasion-induced release.



4 Terminology

Related Documents

ASTM C 153-07 - Abrasion Resistance of Dimension Stone Subjected to Foot Traffic Using a Rotary Platform, Double-Head Abraser

ASTM D 4060-95:2007 - [Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser](#)

DIN 53754:1977 - Testing of Plastics; Determination of Abrasion, Abrasive Disk Method

DIN 68861-2:1981 - Abrasion Resistance

ISO 5470-1:1999 - Rubber- or plastics-coated fabrics -- Determination of abrasion resistance - Part 1: Taber abrader

ISO 15900:2009 - Determination of particle size distribution-Differential electrical mobility analysis for aerosol particles

ISO/CD 27891:2012 - Aerosol particle number concentration-Calibration of condensation particle number counters

ISO/TS 27687 - Nanotechnologies -- Terminology and definitions for nano-objects -- Nanoparticle, nanofibre and nanoplate

Definitions

Abrasion - wearing away: the process of wearing away by friction.

Abrader - wear testing instrument to evaluate abrasion resistance; also referred to as an abraser.

Abrasion cycle - in abrasion testing, one or more movements of the abradant across a material surface, or the material surface across the abradant that permits a return to its starting position. In the case of the rotary platform test method, it consists of one complete rotation of the specimen.

Index of Abrasion resistance - a number calculated from the weight loss of a specimen subjected to a given number of revolutions against a standard bonded abrasive wheel.

Nanomaterials - objects with one, two, or three dimensions in the size range of 1-100 nm.

Nanoparticle - objects with all three dimensions smaller than 100 nm.

Nano-scale - size range of approximately 1nm to 100 nm.

Resurface - procedure of cleaning and refreshing the running surface of an abrasive wheel prior to use in testing.

5 Materials and Apparatus

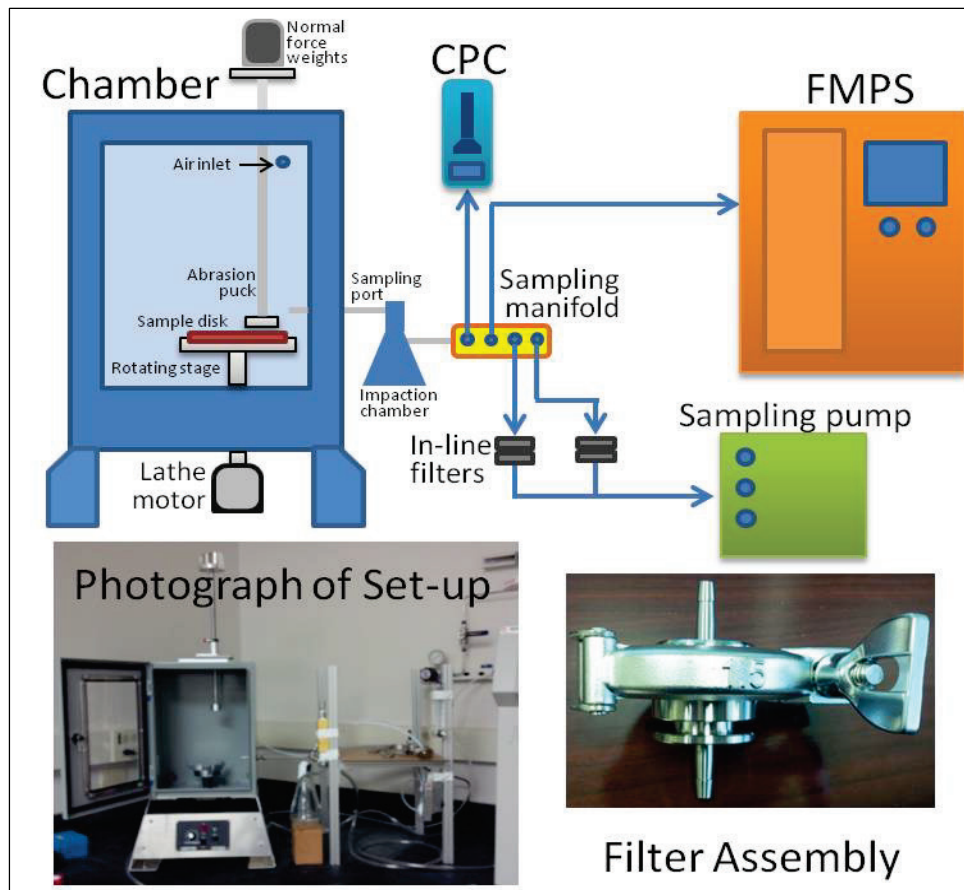
Materials

- Abrasion chamber with controlled, HEPA-filtered air inflow connected to an instrument bank (CPC, FMPS) and in-line filters to collect abrasion-generated particles
- Test samples sized to fit in rotating sample holder (13 cm diameter)
- Adhesive-backed sandpaper of various roughnesses to place on surface of sanding disc
- Weights to use on sanding disc stand to create desired normal force (1-5 kg)
- Isopropanol: For CPC instrumentation
- Supplies for cleaning chamber (vacuum, wipes, water)
- HEPA-filtered air circulation system in the room to reduce background particles and remove any generated particles from the air in the room

Apparatus

Refer to Figure 2 for general experimental setup. A 12 inch x 8 inch x 16 inch cabinet houses the abrasion testing system, which includes a commercial lathe with attached sample-holder, an aluminum disc with replaceable sandpaper attached to the face contacting the sample at the end of a rod with platform for adding mass (to increase normal force during abrasion), inlets for introduction of HEPA-filtered air into the cabinet, and outlets for particulate sampling/collection. The inlet airflow is controlled to 20 lpm, matching or slightly exceeding the flow out to the instruments and collection filters. For the experimental setup shown in Figure 2, the CPC has a flowrate of 0.7 lpm, the FMPS has a flowrate of 10 lpm, and the two in-line filters have flowrates of 4.5 lpm each, for a total sampling rate of 19.7 lpm. The airborne particles from the cabinet are passed through a large impaction chamber to allow settling of large particles, then into a 4-way distribution manifold that splits the flow to the instruments and filters.

Figure 2. Experimental setup for sanding simulation system and sampling monitoring system.



- CPC: for measuring the total number of particles from 10-1,000 nm
- Disc sander: for abrading samples into particles
- Disc plate: for sample mounting to spindle of lathe
- Electric motor: for rotating the disk plate
- HEPA filtered air blower: for maintaining a low background concentration outside the cabinet
- Lathe: for rotating the test sample exposed to the disc sander
- Plastic enclosure: for a secondary enclosure to place around sand blasting cabinet
- Pressure gauge: for monitoring the pressure inside unit
- Pulley: for controlling speed of disk plate
- PVC pipes: for isolating the cabinet from background air
- OPC: for measuring particle number concentrations in channels
- Sand Blasting Cabinet: for housing the disc sander
- SMPS: for measuring particle number channels
- Sampling exhaust pump: for extracting a portion of the cabinet exhaust
- Tachometer: for measuring disc speed and position of the pulley

6 Procedure

- Turn on the room HEPA filter, CPC, FMPS, filtered air inlet, and sample pump.
- Set adjustable disc sander speed (e.g., slow (586 RPM), medium (1425 RPM), or fast (2167 RPM)).
- Mount the test sample on the carriage, and ensure that it is level and that the surface stays level when the motor is engaged and the sample is spinning.
- Connect the zero filter to the CPC air inlet and verify the total particle count is less than 5 particles/ cm^3 .
- Connect the zero filter to the FMPS and reset the zero if necessary.
- Connect the CPC, OPC, and SMPS to sampling manifold.
- Ensure that the filter casings contain appropriate filters (0.2 μm , gold coated for SEM analysis).
- Switch on the computers and ensure connection to instruments.
- Run test:
 - Background collection (60s)
 - Abrasion (240s)
 - Return to background (60-600s)
- Turn off pumps and remove filters for further analysis.
- Collect abraded material from chamber interior for further analysis (optional).
- Vacuum out chamber and wipe with clean, damp wipes to return to clean initial state.

Specimen Preparation

Sample preparation will vary depending on the product selected, but generally they will be prepared by the manufacturing specifications (i.e., epoxy test samples, CNT reinforced epoxy test samples, commercially available CNTs, concrete disks, plates painted with ENM-containing coatings, etc.). All samples should be able to be secured in the sample holder to allow rotation in the chamber. All measurements should be performed in triplicate.

Analysis

CPC and FMPS:

- Launch the control program on the computer, and verify the instrument is communicating with the software.
- Open a new file and run the program (for both instruments) during background collection, abrasion, and return to background.
- Stop the program once the chamber returns to background.
- Export the data as an excel file to an appropriate folder for transfer to external computer for further analyses.

Filtered material:

- After the test concludes, turn off the sample pump that pulls air through the filters.
- Open the filter housings and carefully remove the filter from the support.
 - Care should be taken to not jostle the filter excessively during removal, so as to avoid loss of material into the air. Some filter materials will stick to the support, requiring extra care during removal.
- Place the filter in a container for storage and/or transport for further analysis.
- Further analysis could include SEM imaging, resuspension for chemical/particulate analysis, etc. (See procedure entitled “Quantifying Nanoparticle Release from Nanotechnology ”)

Note: depending on the material, abrasion can remove large amounts of material that can accumulate in the interior of the chamber. This material can be collected for additional analysis as well if desired.

7 Reporting

Analysis of Results

The precision test should be used to examine the consistency of the test run data, including total particle concentration and particle size distribution. Replicate materials can be compared to calculate the mean concentration of total particles and particles at each size distribution. As noted previously, previous studies have shown significant variations between replicates due to differences in microscopic surface topography and smoothness, uneven surfaces or sample mounting, accumulation of abraded material on the sandpaper surface, melting of the sample, or other factors. It is recommended to run at least three tests on each material to help identify the presence of outlier abrasion tests and establish a more confident representation of the release of each material. These multiple comparisons using Tukey's test at $\alpha=0.05$ (significant level) will be performed to compare the difference in number and distribution concentration mean among all test conditions.

Key Results Provided

The key results will be summarized by figures that will show total particle concentrations and particle size distributions. An example of a total released particle concentration is shown in Figure 3 and an example of a particle size distribution is shown in Figure 4. The spread in the data will be shown using error bars or ANOVA values presented in the figure captions. Particle size distribution will be especially helpful in comparing the released material to the expected nanoparticle size, for example, highlighting the release at the 30 nm channel of the FMPS to show possible release of 30 nm TiO₂ particles from self-cleaning concrete.

Figure 3. Total particle concentration released during abrasion of an ABS 3-D printed puck with Bronze nano-colorants, as measured by CPC. Abrasion started at $t=60$ s and concluded at $t=300$ s. Solid line shows average particle counts with thin lines representing \pm standard deviation from three replicate tests.

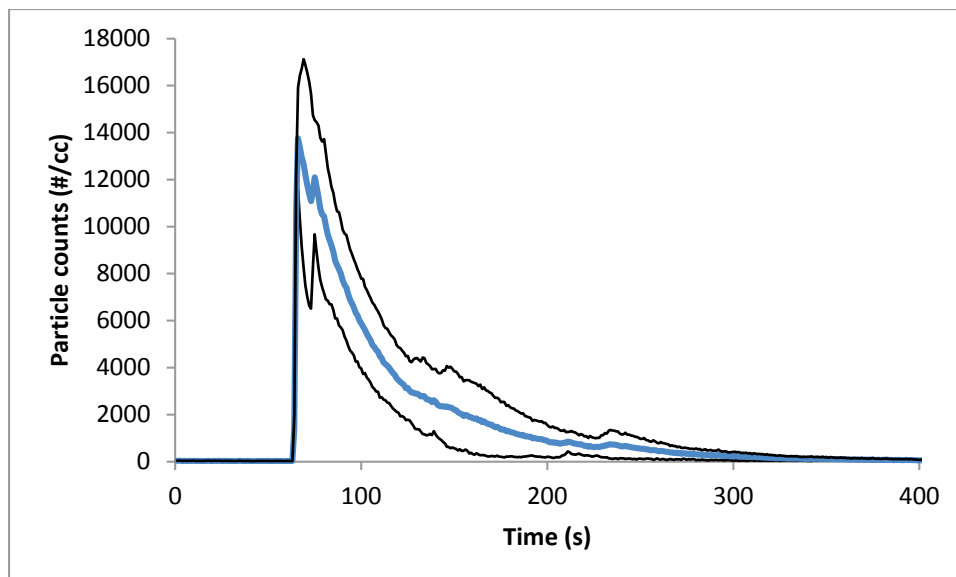
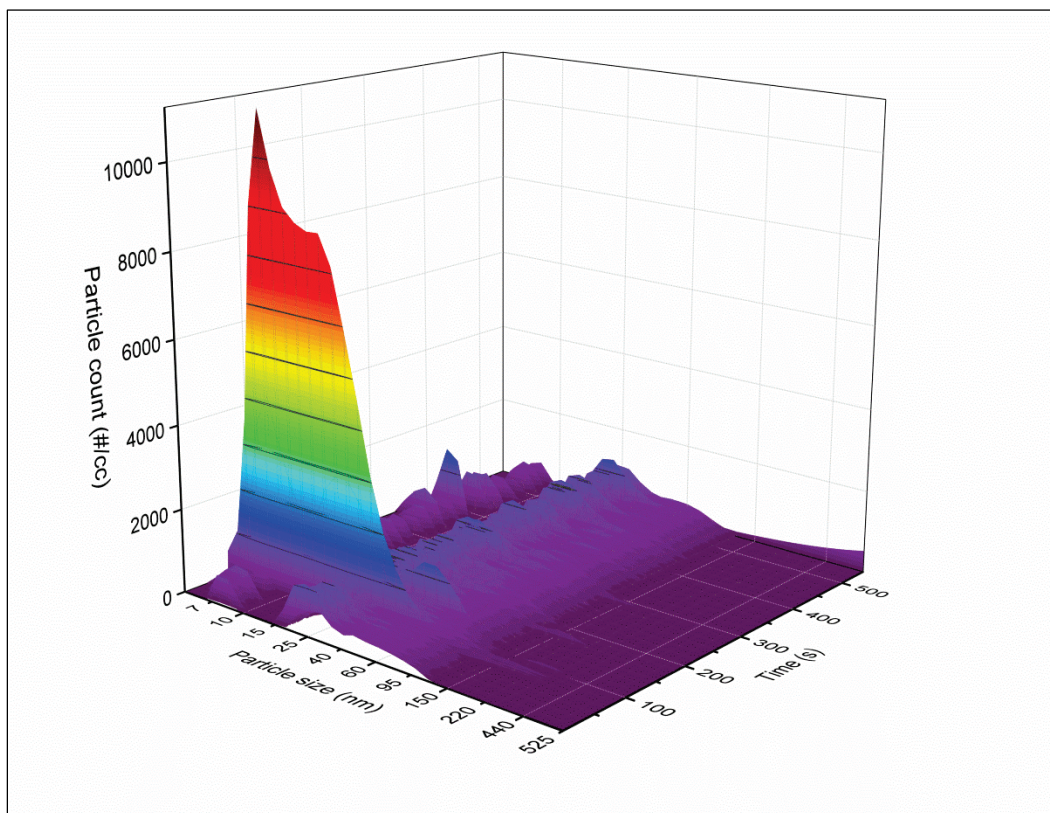


Figure 4. Particle size distribution for abrasion of an ABS 3-D printed puck with nano-colorants, measured by FMPS. Data shows that the majority of particles fall in the 5-50 nm size range.



QA/QC Considerations

Method blanks should be considered for the sample and analysis with sanding simulator running with no abrasion, and with the sandpaper in contact with a non-abrading surface to determine the possible particle contribution of the rotation motor, sandpaper, and residual materials in the system. Also a negative control should be run using the same polymer matrix without nanomaterials. All instrumentation (SEM, CPC, etc.) should follow standard calibrations.

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Appendix: Notes and Supplementary Data

Equation 1.

$$M_R = \frac{\pi}{6} d_{CPC}^3 \rho N S_R(d_{CPC}) + \sum_{i=1}^{15} \pi \rho d_{mid,i}^3 N_{OPC,i} S_R(d_{mid,i})$$

M_R = Respirable Mass Concentration

d_{CPC} = midpoint diameter of the CPC data

ρ = the particle density (assumed to be 1000 kg/m³)

N = number concentration indicated by the CPC

S_R = Function for the fraction of respirable mass

$d_{OPC,i}$ = midpoint diameter of the OPC channel, i

$N_{OPC,i}$ = number concentration indicated by the OPC for a given size channel, i

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